OFFICE OF NAVAL RESEARCH

FINAL REPORT AND

PUBLICATIONS/PATENTS/PRESENTATIONS/HONORS/STUDENTS REPORT

15 July 1988 to 14 January 1992

for

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Force and Tunneling Microscopy Studies of

Atomic Level Mechanical Properties of Solids

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Part I

Note: All work listed here has been performed on a cost sharing basis between IBM and ONR

A. Papers Submitted to Refereed Journals (and not yet published)

B. Papers Published in Refereed Journals

"Measurement of Micromechanical Properties Using a Bidirectional Atomic Force Microscope with Capacitative Detection," G. Neubauer, S. R. Cohen, and G.M. McClelland, in *Interfaces Between Polymers, Metals, and Ceramics*, eds. B. M. DeKoven, A. J. Gellman, and R. Rosenberg, *Mat. Res. Soc. Symp. Proc.* 153, 307 (1989).

"Nanomechanics of a Au-Ir Contact Using a Bidirectional Atomic Force Microscope," S. R. Cohen, G. Neubauer, and G. M. McClelland, *J. Vacuum Sci. and Technol. A* **8**, 3449 (1990).

"Force Microscopy with a Bidirectional Capacitance Sensor," G. Neubauer, S. R. Cohen, G. M. McClelland, D. Horne, and C. M. Mate, Rev. Sci. Instrum. **61**, 2296 (1990).

"Nanotribology of Diamond Films Studied by Atomic Force Microscopy," G. Neubauer, S. R. Cohen, G. M. McClelland, and H. Seki, in *Thin Films: Stresses and Mechanical Properties II* eds. M. F. Doerner, W. C. Oliver, G. Pharr and F. R. Brotzen. *MRS Symposium Proceedings*,**188** (1990), 219-224.

"Diamond Force Microscope Tips Fabricated by Chemical Vapor Deposition," G. J. Germann, G. M. McClelland, Y. Mitsuda, M. Buck, and H. Seki, *Rev. Sci. Instrum.* **63**, 4053 (1992).

"Atomic Scale Friction of a Diamond Tip on Diamond (100) and (111) Surfaces," G. J. Germann, S. R. Cohen, G. Neubauer, G. M. McClelland, H. Seki, and D. Coulman, *J. Appl. Phys.* **73**, 163 (1993).

"Observing the Motion of a Single Surface Atom with Picosecond and Sub-Nanometer Resolution," H. Heinzelmann, F. Watanabe, and G. M. McClelland, *Phys. Rev. Lett.* **70**, 3611 (1993).

"Real-time Observation of the Vibration of a Single Adsorbed Molecule," F. Watanabe, G. M. McClelland, H. Heinzelmann, *Science* **262**, 1244 (1993).

C. Books (and sections thereof) submitted for Publication.

D. Books (and sections thereof) Published.

"Tribology at the Atomic Scale," G. M. McClelland and S. R. Cohen, in *Chemistry and Physics of Solid Surfaces VIII*, eds. R. Vanselow and R. Rowe (Springer, Berlin, 1990), p. 419.

"Friction at the Atomic Scale," G. M. McClelland and J. N. Glosli, in *Fundamentals of Friction: Macroscopic and Microscopic Processes* , eds. I. L. Singer and H. M. Pollock, (Kluwer, Dordrecht, 1992), p. 405.

E. Technical Reports Published and Papers Published in Non-Refereed Journals

The papers listed above are also IBM Research Reports

F. Patents Filed.

G. Patents Granted.

H. Invited Presentations at Topical or Scientific/Technical Society Conferences

"The Atomic Dynamics of Friction," Gordon Conference on Solid State Studies in Ceramics," August 16, 1988.

"Force Microscopy Studies of Friction and Wear," at "Adhesion and Friction: Microscopic Concepts," Halifax, August 25, 1988.

"Atomic Force Microscopy and the Mechanical Properties of Interfaces," Seventh Annual Symposium on Advances in Microscopy, September 24, 1988.

"Direct Observation of Friction at the Atomic Scale," ACS National Meeting, September 28, 1988.

"Direct Observation of Friction at the Atomic Scale," AVS National Meeting, Atlanta, October 6, 1988.

"Atomic Force Microscopy and Friction at the Atomic Scale," after dinner talk, AVS Detroit Chapter, October 14, 1988.

"Direct Observation of Friction at the Atomic Scale," MRS Fall Meeting, Boston, November 30, 1988.

"The Atomic Dynamics of Friction," Israel Chemical Society Annual Meeting, Weizmann Institute, February 6, 1989.

"Measurement of Micromechanical Properties Using a Bidirectional Atomic Force Microscope with Capacitative Detection," G.Neubauer (speaker), S. R. Cohen, and G. M. McClelland, MRS Meeting, San Diego, April 27, 1989.

"Friction at the Atomic Scale," New Mexico AVS Meeting, Albuquerque, May 11, 1989.

"Friction at the Atomic Scale," ACS Meeting, April 10, 1989.

Tribology at the Atomic Scale," Eighth International Summer Institute on Surface Science, Milwaukee, August 22, 1989.

"Tribology at the Atomic Scale" Sandia National Labs, February 9. 1990.

"The Atomic Dynamics of Friction," Gordon Conference on Energetic Material, June 1990.

"The Atomic Dynamics of Friction," International Symposium on Industrial Tribology, Chicago,

"Friction at the Atomic Scale," West Coast Spectroscopy Conference, Asilomar, February, 1991.

Tribology at the Atomic Scale," March APS meeting, Cincinnati, 1991.

Lecture at NATO ASI on Fundamentals of Friction, "Friction at the Atomic Scale," Braunlage, Germany, July, 1991.

I. Contributed Presentations at Topical or Scientific/Technical Society Conferences

S. Cohen (speaker), G. Neubauer, and G. M. McClelland, "Nanotribology Using a 2D AFM with Capacitative Detection," AVS Meeting, Boston, October 26, 1989.

"Fundamental Studies of Friction Using a Bidirectional UHV Force Microscope," G. M. McClelland, G. J. Germann, G. Neubauer, and S. R. Cohen, International Conference on Metallurgical Coatings and Thin Films, San Diego , April, 1991.

"Bidirectional AFM Study of Diamond-Diamond Friction in UHV," International STM Conference, Interlaken, August, 1991.

"Bidirectional AFM Study of Diamond-Diamond Friction in UHV," AFOSR contractors meeting, Irvine, October 22, 1991.

J. Honors/Awards/Prizes

Gary McClelland was named an American Physical Society Fellow in October, 1990.

K. Number of Graduate Students Receiving Full or Partial Support on ONR Contract

zero

L. Number of Postdoctoral Fellow Receiving Full or Partial Support on ONR Contract

two

Part II

A. Principal Investigator

Gary M. McClelland

B. Cognizant ONR Scientific Officer

Dr. Richard Brandt

C. Current telephone number

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D. Brief Description of project

We are using atomic force microscopy (AFM) to characterize the tribological properties of materials on an atomic scale. For these experiments we employ an ultrahigh vacuum instrument which measures the lateral and normal forces and current through a tip in contact with a surface which can be positioned with sub-Angstrom resolution. Friction of a sharp tip on a surface is investigated by profiling the surface, sliding the tip across the surface while measuring the normal and parallel forces and contact resistance, and profiling the surface after sliding.

E. Significant results

Under this project the construction of the first ultrahigh vacuum atomic force microscope (AFM) was completed. This same instrument was the first AFM to employ capacitance for measuring the deflection of the force-sensing cantilever and was also the first AFM to measure forces simultaneously both parallel and perpendicular to the sample surface, a practice now adopted by many other groups. Electronics for fast, sensitive and drift-free capacitance measurement were developed, and the noise sources for the capacitance technique were analyzed in detail.

The bidirectional vacuum AFM was used to study the tribology of an iridium tip on Au(111) in UHV. When it was published, this was the highest resolution tribology study ever performed on metals. Elastic indentation experiments only 10 Å deep into the surface can be interpreted in terms of Angstrom-scale surface roughness. Friction loops 50 Å wide observed at $\simeq 10^{-7}$ N were free of any stick-slip structure associated with a difference between static and dynamic friction or with any features on the surface. Sliding the surface back and forth across the tip while advancing it toward and away from the tip, we found that the friction changed much more rapidly with increasing load (effective friction coefficient = 1.0) than with decreasing load (effective friction coefficient = .4). This difference appeared, even though any plastic deformation during contact was no more than 1 Å, indicating that extremely subtle changes in the tip-surface interface must effect the frictional force. In the friction loops, a curvature is observed just before full sliding, an effect attributed to partial stick/partial slip of the tip.

A CVD method was developed for forming single crystal diamond force microscope tips. By examining the tips by SEM at very low electron dose and thermally flashing in UHV, tips of known orientation and surface chemistry were

prepared. There are the best-characterized tips to have been used in AFM experiments.

Using the diamond tips, we investigated diamond-diamond friction in UHV. The measured attractive tip-surface interaction agreed with predicted dispersion interactions. On the (111) surface, the frictional force shows features arising from the (1×1) reconstructed hydrogen-terminated crystal surface. The lateral force appears to be the sum of a position-dependent conservative part and a position-independent dissipative part, which changes sign with the direction of motion. The differential friction coefficient is nearly zero, suggesting the interface may be in the regime of "zero friction" predicted for incommensurate weakly interacting solids.

As a method to characterize tips, we became interested in field ion and field emission microscopy. This led to the development of the femtosecond field emission camera (FFEC), a device for observing the dynamics of individual atoms and molecules on the subpicosecond time scale. In this instrument an electron beam field-emitted from an extremely sharp metal tip is focussed and swept across a detector screen. The motion of a single adsorbed atom on the tip causes a fluctuation in the field emission which is recorded on the screen. During the ONR contract, only the construction of this instrument was completed. Subsequent work has recorded with 1 ps resolution the hopping of single Cs atoms between sites on a tungsten tip and the vibration of single adsorbed copper phthalocyanine molecules with respect to the tip.